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1. REPORT DATE (DD-MM-YYYY) 07-02-2005		2. REPORT TYPE Final Report		3. DATES COVERED (From – To) 8 February 2004 - 08-Feb-05	
4. TITLE AND SUBTITLE An Investigation into the Effect of Stabiliser Content on the Minimum Characteristic Chamber Length for Homogeneously-Catalysed Hydrogen Peroxide			5a. CONTRACT NUMBER FA8655-04-1-3002		
			5b. GRANT NUMBER		
			5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Dr. Antony J Musker			5d. PROJECT NUMBER		
			5d. TASK NUMBER		
			5e. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) DELTACAT Limited 4 Ryde Place Lee on Solent PO13 9AU United Kingdom				8. PERFORMING ORGANIZATION REPORT NUMBER N/A	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) EOARD PSC 802 BOX 14 FPO 09499-0014				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) SPC 04-3002	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES					
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15. SUBJECT TERMS Hydrogen peroxide, high test peroxide, HTP, rocket grade hydrogen peroxide, RGHP, stabilized hydrogen peroxide, EOARD.					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18, NUMBER OF PAGES 37	19a. NAME OF RESPONSIBLE PERSON BARRETT A. FLAKE
a. REPORT UNCLAS	b. ABSTRACT UNCLAS	c. THIS PAGE UNCLAS			19b. TELEPHONE NUMBER (Include area code) +44 (0)20 7514 4285

Final Report, Dated January 2005, Covering Grant Number FA8655-04-1-3002, Awarded by the Air Force Office of Scientific Research, Air Force Material Command, USAF, and Administered by EOARD (London).

An Investigation into the Effect of Stabiliser Content on the Minimum Characteristic Chamber Length for Homogeneously-Catalysed Hydrogen Peroxide

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Executive Summary

This report assesses the extent to which chemical stabilisers added to hydrogen peroxide affect the peroxide's ability to decompose rapidly in the presence of a liquid catalyst. The catalyst used was sodium permanganate monohydrate at a nominal specific gravity of 1.35. A small decomposition chamber was constructed in accordance with a simple steady-state, thermo-chemical model of the process. The hydrogen peroxide concentration varied between 81% and 83% and covered a range of phosphorus content between 0.3 and 300 parts per million by mass. The stagnation temperatures along the length of the chamber were monitored to try to determine when decomposition was complete, as judged by comparison with the theoretical values calculated from the thermo-chemical model. The tentative conclusion reached is that for this method of catalysis the stabiliser content does not affect the rate of peroxide decomposition.

ABSTRACT

Rocket engines that use rocket-grade hydrogen peroxide (RGHP) normally rely on solid catalyst packs to achieve a heterogeneous catalytic reaction to decompose the propellant into steam and oxygen. The decomposition of highly stabilised hydrogen peroxide (HSHP) using a liquid catalyst offers an alternative to this approach. HSHP offers the prospect of longer-term storability and ease of handling compared with the relatively unstabilised rocket-grade hydrogen peroxide demanded by catalyst packs. However, one issue that has been hitherto ignored has been the extent to which stabilisers added to the HSHP at source might adversely affect its rapid decomposition. A test rig has been built to address this by attempting to estimate the length of chamber required for complete decomposition to take place. This has been done for the case of HSHP with a phosphorus content some 1000 times the level normally associated with rocket-grade material. These results have been compared with those for RGHP at the same concentration. The overall conclusion is that the stabiliser content has no significant effect on the observed decomposition rate.

1. INTRODUCTION

This paper concerns the development and testing of a rig designed to investigate the homogeneous catalytic decomposition of hydrogen peroxide (often referred to as 'high test peroxide', or HTP - here used in a general sense to include either highly stabilised or rocket-grade material). The purpose of the investigation was to determine to what extent the presence of stabilisers in highly stabilised hydrogen peroxide (HSHP) might inhibit the decomposition process. To achieve this, a direct comparison was made between HSHP and rocket-grade hydrogen peroxide (RGHP).

If HTP is injected into a stream of a suitable liquid catalyst at one end of an enclosed, cylindrical, reaction chamber, one would expect the subsequent catalytic reaction to be complete, under ideal mixing conditions, within a certain length of chamber. The main factors that control the idealised decomposition rate are the HTP concentration, the mixing arrangements, the HTP stabiliser content and the catalyst-to-HTP mixture ratio (R). The present study focuses on the third of these and was carried out for a nominally constant HTP concentration. However, before this could be done, a pilot study was

first undertaken to set the propellant injectors and to determine an appropriate mixture ratio for the duration of the investigation. Low mixture ratios lead to incomplete reactions within a finite length of chamber. On the other hand, high ratios guarantee rapid reaction within a short advection length at the expense of unacceptably low decomposition temperatures on account of the excessive heat lost associated with vapourising the catalyst solution.

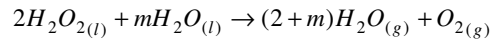
The study was conducted in two parts. The first part (Phases 1 and 2) concentrated on pilot runs aimed at identifying a suitable mixture ratio. This ratio was then kept constant for the second part (Phase 3), which focused on comparing the behaviour of highly stabilised and rocket-grade forms of HTP.

This report starts with an account of a simple thermo-chemical model of the process within the reaction chamber. The test rig is then described and this is followed by a description of the method used to manufacture the different types of HTP. The next two sections deal with the pilot study, to determine an appropriate mixture ratio, and the final study, to determine the effect of the stabiliser content on the HTP decomposition process. The results are presented separately. The report concludes with a discussion of the results followed by recommendations for further work.

2. THERMO-CHEMICAL MODEL

A simple thermo-chemical model of the decomposition of hydrogen peroxide by homogeneous catalysis was developed. The purpose of the model was twofold: to act as a design tool and to predict the performance of the actual test rig. When used as a design tool the main aim was to determine a range of appropriate catalyst injector diameters and to evaluate the temperatures expected as a result of the decomposition reaction.

The basis of the model is the decomposition reaction:



for which the heat of decomposition per unit mass of hydrogen peroxide is known. It can be observed that the catalyst, a near-saturated (approximately 50%) aqueous solution of sodium permanganate, does not feature explicitly in the equation. However, it does feature indirectly through the second term, which accounts for the excess water in the system. This term also reflects the fact that the hydrogen peroxide is not pure but is itself in an aqueous solution. When using the model as a design tool, the concentrations of the catalyst and peroxide solutions, and hence the amount of excess water, could be set within a spreadsheet.

Inclusion of this excess water is critical to the accuracy of the model, since not only is the overall mass of the reactants (and products) increased, but latent heat is required to convert this excess water from the liquid to the gaseous state. Consequently the predicted decomposition temperature, T_c , is a strong inverse function of the mixture ratio. Furthermore, as is to be expected, for small mixture ratios the effect of the excess water is weak and the decomposition temperature then tends to be dominated by the selected HTP concentration. This suggests that if R can be kept low enough, and the HTP concentration high enough, then a future bi-propellant engine could be made to ignite spontaneously by the present mechanism of homogeneous catalysis.

A recognised weakness of the present thermo-chemical model is the lack of chemical kinetics. Thus the length of chamber available for the reaction to proceed to completion is assumed to be infinite, whereas in practice the required length is finite and defines the so-called characteristic chamber length, or L^* . In addition, the chamber is assumed to be perfectly insulated from heat losses. Nevertheless the model does provide an indication of the likely asymptotic limit associated with the approach to complete reaction. Further development of the thermo-chemical model to incorporate reaction kinetics was beyond the scope of the present study.

By invoking mass continuity and assuming that the decomposition products behave as an ideal gas, the exit diameter of the reaction chamber was calculated as a function of the mixture ratio, assuming that the flow was sonically choked at the exit. Also, for a given chamber stagnation pressure, the HTP injector size could be determined. This was kept constant, but the catalyst injector diameters were allowed to vary to control the mixture ratio.

One of the constraints imposed on the investigation concerned the desire to reduce the amount of HTP needed to a practicable minimum (see Section 5). Consequently, the model's associated spreadsheet

was used to explore various options, from the standpoint of designing the rig, with this constraint in mind. This led to the following choice for some key design parameters:

Nominal tank delivery pressure: 15 bar gauge

Chamber internal diameter: 10 mm

HTP injector internal cross-sectional area: 3.14 mm^2

catalyst injector diameter (range): 0.3 – 1.0 mm

mixture ratios (range): $0.3\% \leq R \leq 30\%$

The predicted decomposition temperatures associated with the above parameters were found to be in the range $207 < T_c < 516 \text{ deg C}$, depending on the HTP concentration.

3. THE TEST RIG

DELTACAT Ltd runs a small test-site in Hampshire, England. Recently, DELTACAT has made this available to selected students who are participating in an accredited Master of Engineering (MEng) degree programme at the University of Southampton. An obvious requirement is that such investigations must be undertaken with very close supervision. Safety of design and operation was therefore critical and this led to large structural safety factors being applied to the system.

After considering various options for mixing the liquids, the arrangement shown in CAD form in Figures 1a and 1b was chosen. This comprised a single, axial catalyst injector, which screwed into the HTP injector manifold. Figures 2 and 3 show the actual components prior to installation in the rig. The manifold comprised a feed pipe to an annular plenum chamber, which, in turn, fed four angled injector holes. The catalyst and HTP streams were designed to impinge on the central axis, 5 mm downstream of the chamber flange connecting the injector head to the reaction chamber. The latter components were separated by a graphite-based gasket and a urethane sealant to minimise heat loss. The impingement point of the catalyst and HTP streams was subsequently used as a datum for determining axial temperature profiles.

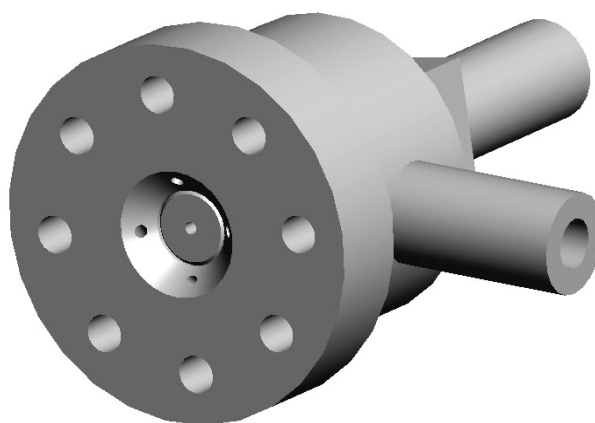


Figure 1a. Injector Head Assembly

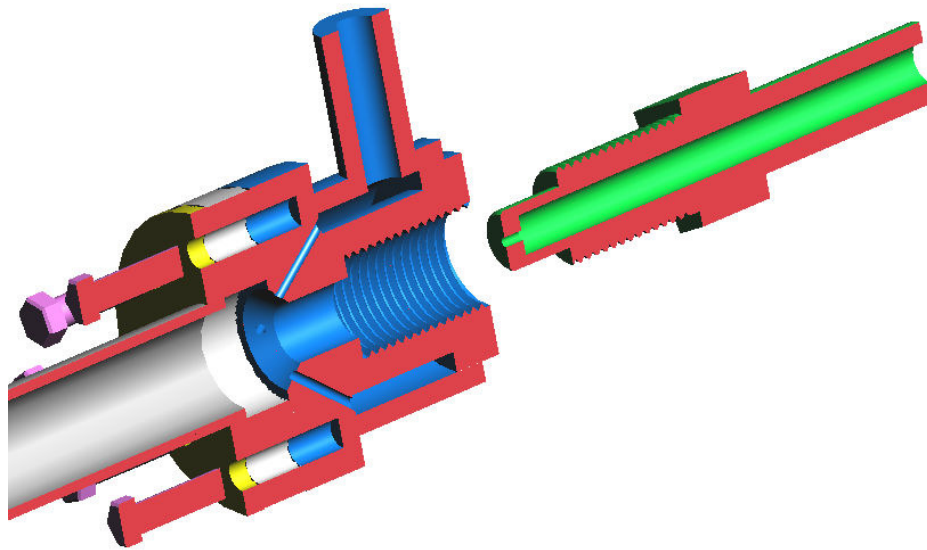


Figure 1b. Injector Head Assembly



Figure 2. Typical Catalyst Injector (0.7 mm Orifice)

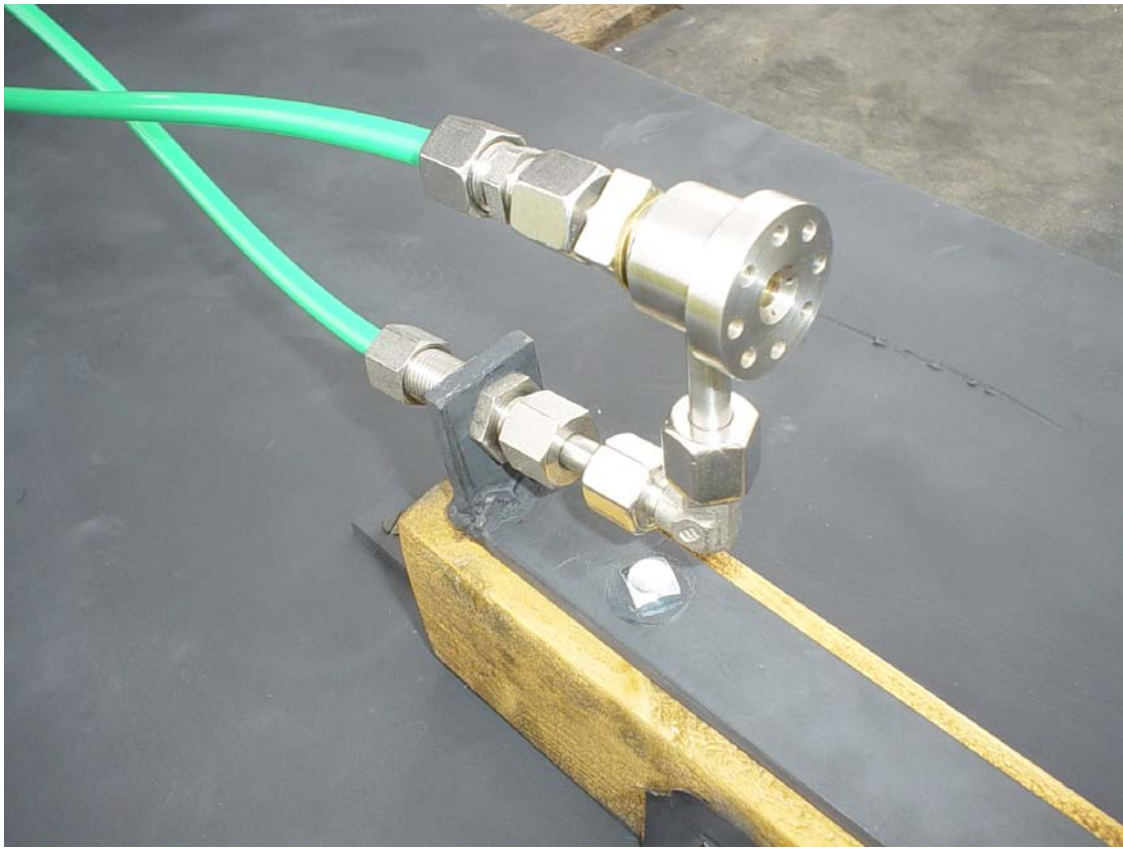


Figure 3. Manifold Assembly

The injector head was manufactured from 316 stainless steel, as were some of the catalyst injectors (the smaller diameter injectors were made of brass for ease of manufacture). The reaction chamber itself was 500 mm in length, and was constructed using 316 stainless steel tube with a 1 mm wall thickness and 10 mm internal diameter. The chamber was fitted with a simple divergent conical nozzle designed to match atmospheric pressure at the exit plane (Figure 4). Note that the contraction ratio of the chamber was unity. For Phases 1 and 2, stainless steel-sheathed, 1.5 mm diameter mineral-insulated type K thermocouples were brazed through the wall of the reaction chamber at stations 10 mm, 100 mm and 450 mm downstream of the datum (impingement) point to determine the axial temperature profile. For the Phase 3 runs, additional thermocouples were placed at 50 mm and 200 mm downstream of the datum point. In all cases the thermocouple tips were aligned along the chamber radii. The thermocouple brazing material used was cadmium-free Alloy 424 (Ag 24%, Cu 43% and Zn 33%) with a melting point in excess of 740 degrees Centigrade.

A photograph of the overall chamber assembly is shown in Figure 5. For Phases 1 and 2 the thermocouple signals were recorded on a PC via an 11-channel analogue to digital converter (ADC) after first being amplified using a four-channel instrumentation grade amplifier. However this system failed after the Phase 1 runs and was replaced with a bank of digital voltmeters set at 200mV range for Phases 2 and 3. The displays were recorded using a Sony Hi-8 camcorder and subsequently digitised with a GRANDTEC module and then read using ULEAD VideoStudio 6 software.



Figure 4. Expansion Nozzle

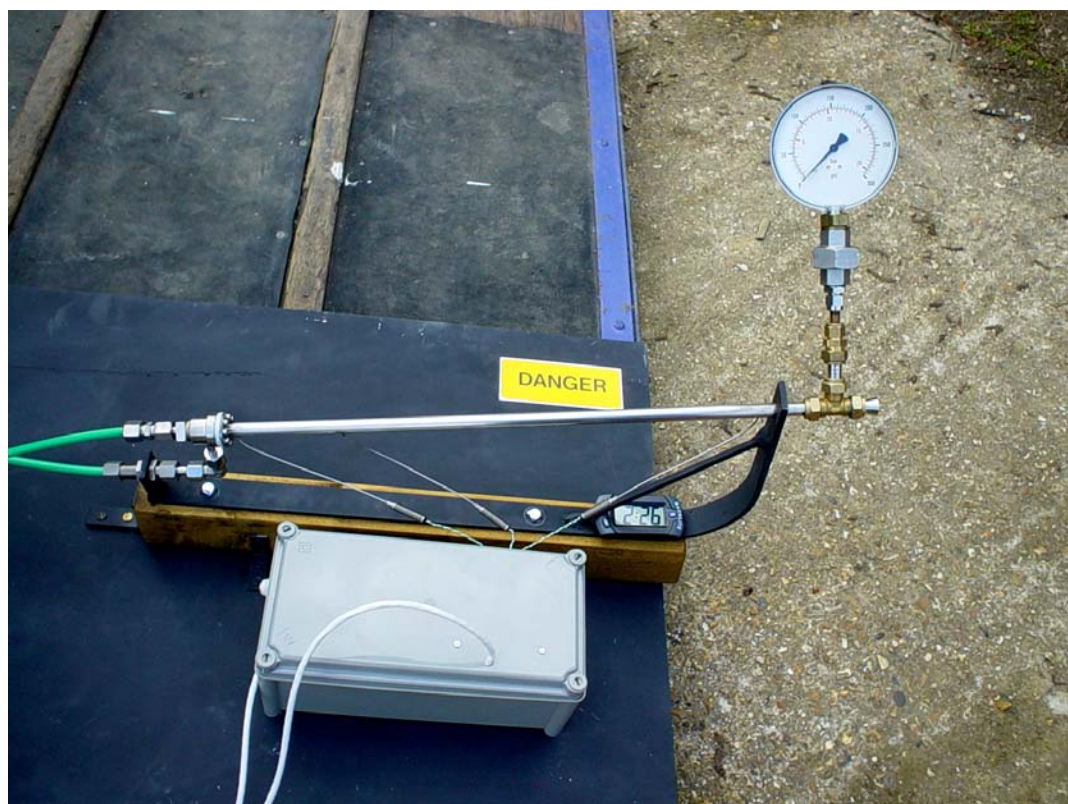


Figure 5. Reaction Chamber Assembly



Figure 6. Propellant Delivery System

The propellant delivery system (Figure 6) incorporated propellant storage tanks, manual shut-off valves, pneumatically-operated ball valves (Figure 7), suitably vented in the case of HTP, and various pilot controls in the form of DC-operated solenoid valves. The latter were operated remotely from a console (Figure 8) to ensure safety of personnel. The propellant feed system was pressurised using dry nitrogen drawn from a regulated 230 bar supply.

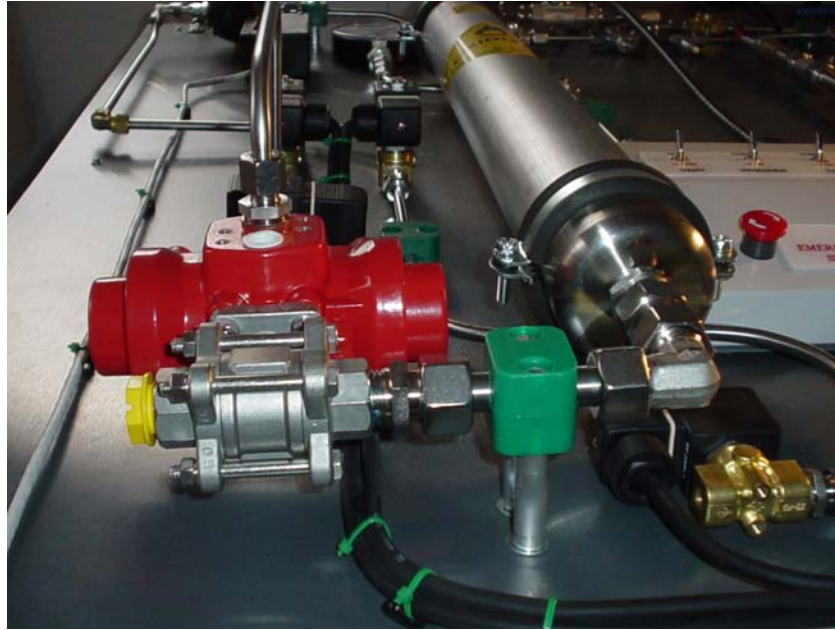


Figure 7. HTP Pneumatically-Controlled Ball Valve



Figure 8. Control Console

4. HTP SUPPLY

Commercial grade hydrogen peroxide (UN2014) is readily available in concentrations up to 50%. This is always heavily stabilised. Food and semi-conductor grades up to 35% can be obtained easily and are associated with very low levels of added stabilisers. However, the concentration of such grades in reasonable quantities should be performed under a low vacuum. Unfortunately rocket-grade HTP

(RGHP, UN2015) at 90% concentration is almost impossible to procure in the UK unless the purchaser is a major government establishment. Ideally, this would be the material of choice, since all that needs to be done to convert it to HSHP is to add the stabiliser(s) to the required level. However, DELTACAT Limited has a long record of successfully producing concentrated HSHP in batches of typically 1 kg in relative safety.

The starting material for Phases 1 and 2 was commercial grade hydrogen peroxide at 50%. Using scrupulously clean equipment, the details of which are proprietary to DELTACAT, this material was gently boiled at atmospheric pressure at a remote test-site. It should be stressed that the apparatus was itself remote from the operator in case of vapour phase detonation. In more than forty hours of operation this has never been witnessed. Nevertheless, and to avoid any risk of such a detonation transferring to the liquid phase, however unlikely, DELTACAT imposes a limit of 84% on the target strength of HSHP. Note that the final concentration was determined according to the measured density at ambient temperature. The uncertainty in concentration is approximately $\pm 0.5\%$, based on a density uncertainty of $\pm 0.7\%$.

For the Phase 3 tests the above procedure is far too dangerous because the starting material, in this case semiconductor grade hydrogen peroxide at 30% concentration, is almost free of stabilisers. This was heated gently at 60 degrees Centigrade in passivated aluminium trays at atmospheric pressure. Although this was a tedious procedure, the net result was a sufficient quantity of material that compares well with rocket-grade hydrogen peroxide in terms of its trace elements. From here on, and for the purpose of this report, the Phase 3 hydrogen peroxide will be referred to as RGHP.

Representative samples from the start and end batches of both HSHP and RGHP were diluted with very pure, demineralised water, weighed before and after the dilution, and then subjected to spectral analysis to identify the quantities of key trace elements associated with likely added stabilisers or contaminants associated with the concentration procedure. The only reason for this dilution was to allow unrestricted road transportation to the testing laboratory. The analysis was performed using an inductively-coupled plasma-optical emission spectrometer. The results, adjusted back to the original undiluted samples, in parts per million by mass (ppm), are shown in Table 1. The low sodium levels detected in relation to phosphorus suggest phosphoric acid, H_3PO_4 , rather than (tetra)sodium pyrophosphate, $Na_4P_2O_7$, as the stabiliser for Phases 1 and 2 (note that, even at the level of senior management, the supplier refused to reveal the nature of the additive(s) used). Certainly stannates, for example sodium stannate, can be ruled out as the stabiliser for all the phases. The Phase 3 stabiliser is thought to be sulphuric acid, with a total sulphate content of less than 1 ppm for the source material.

Table 1. Trace Elements (ppm)

Batch	concentration	Fe	P	Sn	K	Na	Cr	Ni	Al
Phases 1 and 2 starting batch	50%	0.6	60	<0.06	<0.2	1.1	0.2	0.2	-
Phase 1	81%	2	299	<0.15	<0.5	11	0.9	0.5	-
Phase 2	83%	2	299	<0.15	<0.5	11	0.9	0.5	-
Phase 3 starting batch	30%	<0.05	<0.3	-	<0.1	<0.1	<0.02	<0.02	<0.5
Phase 3	83%	0.085	0.28	<0.085	0.57	1.8	-	-	0.85
MIL-PRF-16005F Type 90 Grade HP	90%	<0.03	<0.06	<4	-	<1.9*	<0.03	<0.03	<0.35

*assumes sodium nitrate as additive/stabiliser

Of particular note is the fact that the phosphorus level content in the final concentrated batches for Phases 1 and 2 is one thousand times higher than that found in the Phase 3 unstabilised material. The complete MIL-RGHP specification is available in Reference [4].

5. PILOT STUDY (PHASES 1 and 2)

Because of the students' involvement in the test programme, all DELTACAT's site arrangements needed the approval of the University of Southampton's Health and Safety Officer. Accordingly, the present tests were planned in great detail and with an emphasis on safety - brought about by conducting the tests according to very detailed procedures (see Appendix 1). The primary aim of Phases 1 and 2

was to commission the rig and to perform preliminary tests before embarking on the Phase 3 experiments.

The first runs were simply spray tests using water. These were followed by several runs using water to determine the injector discharge coefficients. Additional tests to check the logistics and operating procedures were also performed. After minor refinements, the rig was deemed ready for useful work.

Six live runs were planned, using catalyst injectors with the following hole diameters: 1.0, 0.85, 0.7, 0.5, 0.4 and 0.3 mm. According to the thermo-chemical model, this spanned a range in R, the catalyst-to-HTP mixture ratio, or loading factor, from 31% down to 2.9% respectively. All runs were performed with a catalyst concentration of 107 grams of (solid) sodium permanganate monohydrate to 100 cc of demineralised water. When using the thermo-chemical model, a value of 40% water content for the catalyst solution was used, in contrast to the experimental value of 52%, as this accounted, albeit crudely, for the heat capacity of the sodium permanganate in solution.

The aim of each run was to record the time history of the thermocouple temperatures, the tank and chamber pressures and also the visual character of the chamber exhaust plume. After preparing the rig and filling the tanks, all personnel retreated to a control centre located some 50 metres away. The rig was then armed and made live after the Range Safety Officer had given clearance.

6. RESULTS OF PILOT STUDY

Nine runs were performed in total, including three extra runs that had not been planned using a higher strength stock of hydrogen peroxide. Table 2 summarises the run schedule.

Table 2. Phases 1 and 2 Run Schedule

phase	run#	date	HTP %	catalyst injector diameter mm	catalyst loading %	maximum observed steady stagnation temperature deg C	theoretical stagnation temperature deg C	observed chamber pressure bar abs	theoretical chamber pressure bar abs
1	1	8/4/04	81	1.0	31	196	228	*	8.5
1	2	11/4/04	81	0.7	15	379	369	10.7	9.1
1	3	11/4/04	81	0.5	7.7	rejected	444	*	9.2
1	4	11/4/04	81	0.3	2.8	rejected	498	*	9.2
1	5	14/4/04	81	1.0	31	243	228	*	8.5
1	6	14/4/04	81	0.5	7.7	448	444	*	9.2
2	7	9/5/04	83	0.7	15	404	413	11	9.4
2	8	9/5/04	83	0.85	22	388	344	11.5	9.2
2	9	9/5/04	83	0.4	4.9	494	521	*	9.5

* not recorded

Most runs were conducted successfully at a nominal tank gauge pressure of 15 bar. However, the data for Runs 3 and 4 had to be rejected because of intermittent and hesitant behaviour observed in the plume due to a blockage in the catalyst injector. It was found that the thermocouples needed about four to five seconds to reach a more or less equilibrium temperature with the largest catalyst injector. This was just achievable using the full capacity of the catalyst tank (125 ml). The smallest injector resulted in higher temperatures and needed about eight seconds to reach equilibrium. The maximum run time, using the 0.5 mm injector, was 17 seconds (Run 6).

Visually (see Figure 9 - available in monochrome only), the plumes were pale brown with the larger catalyst hole diameters, tending towards pale blue for the smallest size. This is consistent with the decomposition of the permanganate into manganese dioxide caused by the high internal temperature. In this sense the catalyst can be regarded as consumed.



Figure 9. Plume During Run for Catalyst Injector Hole Diameter = 0.85 mm

Figure 10 shows the time histories of the measured stagnation temperatures at the three chamber stations for a catalyst injector hole of 1.0 mm (note that the 450 mm position refers to the station nearest to the nozzle). The uncertainty in the measured thermocouple temperatures is ± 12 deg C based on the instrumentation errors associated with the digital voltmeters.

The plateaux associated with the equilibrium temperatures for each station were used to show how the measured decomposition temperatures compared with the theoretical predictions using the thermochemical model. These are shown in Figs. 11-13. Note that two batches of HSHP, with concentrations of 81% and 83%, were used for the test programme. It seems reasonable to conclude that complete decomposition was achieved at the nozzle station, which was located 450 mm from the point of jet impingement, for all catalyst loadings (Figure 11).

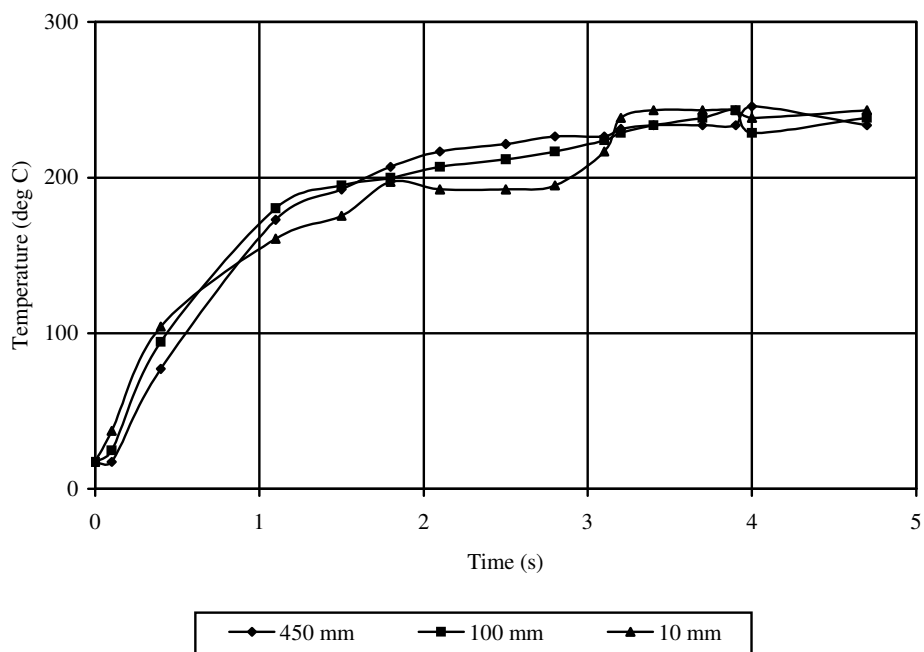


Figure 10. Run 5 Indicated Stagnation Temperatures for Catalyst Injector Hole Diameter = 1.0 mm

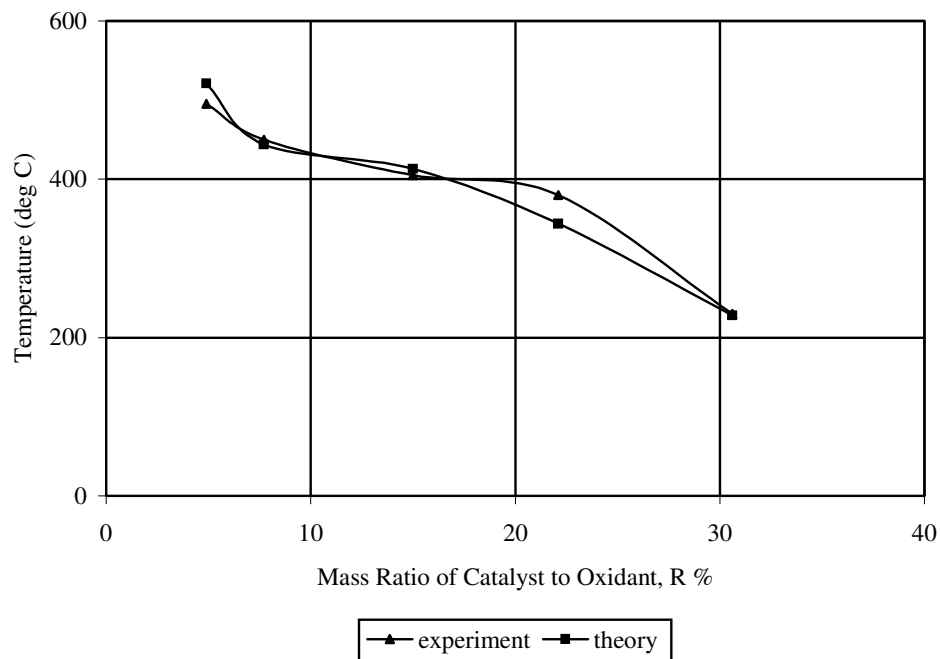


Figure 11. Final Stagnation Temperature 450 mm from Impingement Point

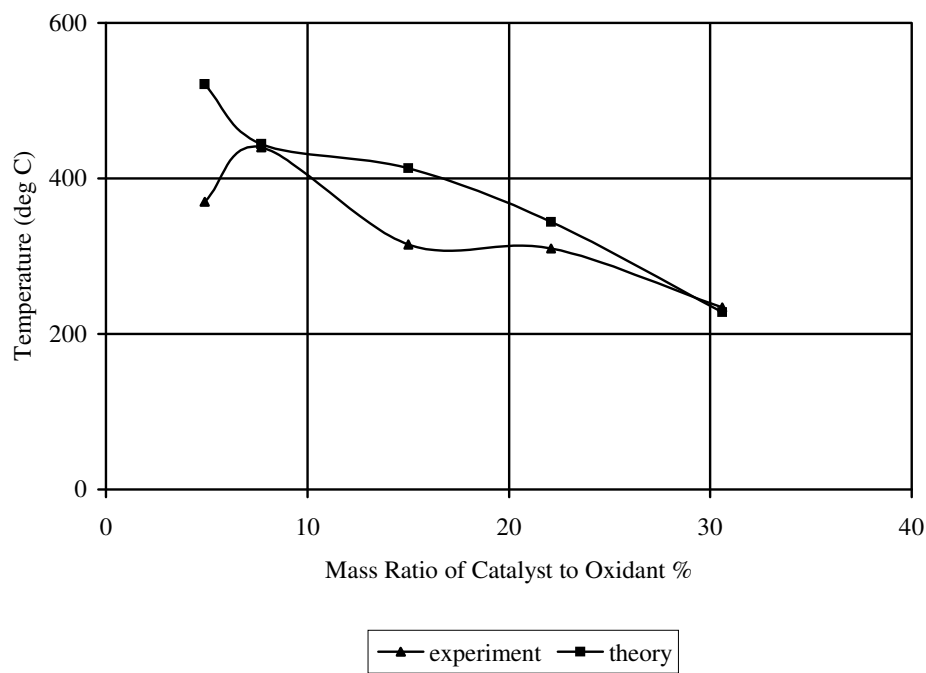


Figure 12. Final Stagnation Temperature 100 mm from Impingement Point

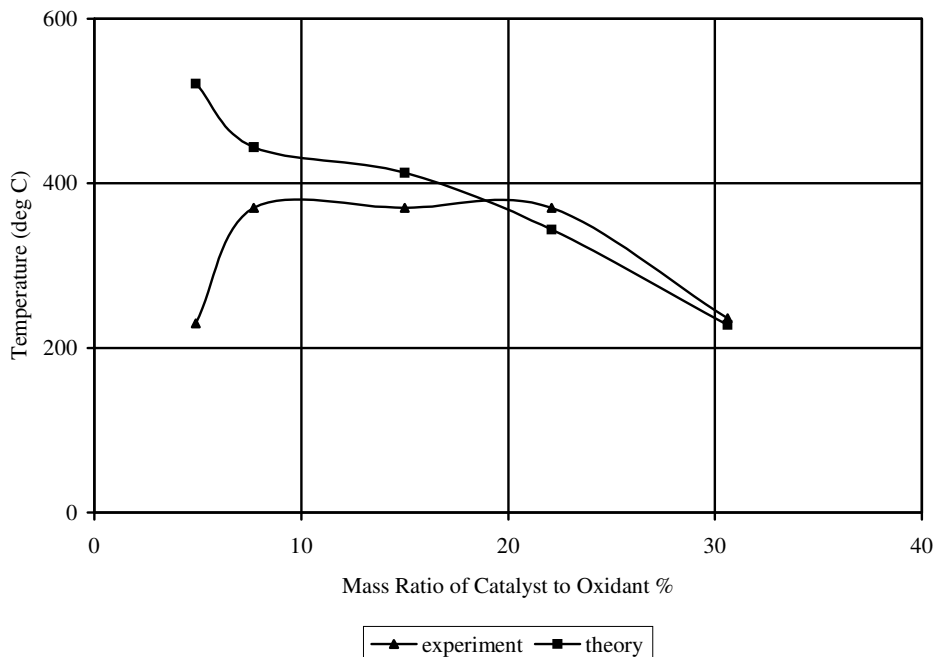


Figure 13. Final Stagnation Temperature 10 mm from Impingement Point

The measured stagnation temperatures at the station 100 mm from the jet impingement point again show reasonably complete decomposition of the HSHP, except for the smallest and intermediate mixture ratios (Figure 12). Decomposition appears to be mostly complete just 10 mm downstream of the injector impingement point for values of $R > 15\%$ (Figure 13). For the smallest injector size, corresponding to $R = 5\%$, the decomposition temperature was approximately one half of the theoretical value, indicating that the flow was still very reactive in this region. Nevertheless these data tend to support the view that the mechanism of homogeneous catalysis of hydrogen peroxide is very effective.

7. EFFECT OF HTP STABILISERS ON DECOMPOSITION (PHASE 3)

The aim of the Phase 3 tests was to identify to what extent the presence of HSHP stabilisers might affect the temperature profiles compared with RGHP. Two batches of HTP were prepared. The first was entirely HSHP and the second was entirely RGHP. Then the batches were adjusted in concentration until they were equal to within half of a percent based on densitometric measurements. Five blended samples, each weighing 686 g, were then prepared for the live runs. The samples were designed to cover the spectrum from 0% percent RGHP (or 100% HSHP) to 100% RGHP (or 0% HSHP). This gave three intermediate blends of 25%, 50% and 75% RGHP. Comparison between the extreme values was regarded as critical as this represented the essence of the overall investigation.

To exercise control over the investigation, all other parameters were held sensibly constant (apart from a 3 degree change in ambient temperature throughout the duration of the Phase 3 tests). The catalyst injector was selected based on the experience gained in Phases 1 and 2. Clearly the smallest injector was unsuitable because of the danger of precipitation in the 300 micron hole. On the other hand the 1.0 mm injector, corresponding to a catalyst-to-HTP mixture ratio (loading factor) of 31%, was considered to be too large since complete decomposition appeared to take place very quickly within the reaction chamber. Accordingly, a catalyst injector diameter of 0.7 mm was chosen as a compromise, giving a theoretical decomposition temperature of 413 degrees Centigrade. This represented a catalyst loading factor of 15%. The Phase 3 tests were conducted in the same manner as Phases 1 and 2 except that the temperatures were recorded at five positions along the longitudinal axis of the reaction chamber instead of three.

8. PHASE 3 RESULTS

The run schedule is shown in Table 3. Runs 1 and 2 - the critical runs - offer a direct comparison between RGHP and HSHP. As Table 2 shows, the phosphorus level for the HSHP propellant was greater than one thousand times the level of the RGHP propellant. Video analysis of Run showed signs of significant blockage and this was confirmed by later inspection of the thermocouple readings. Accordingly the data for Run 4 were rejected.

Table 3. Phase 3 Run Schedule

phase	run#	date	%RGHP	catalyst injector diameter mm	catalyst loading %	maximum observed steady stagnation temperature deg C	theoretical stagnation temperature deg C	observed chamber pressure bar abs	theoretical chamber pressure bar abs
3	1	18/7/04	0	0.7	15	400	413	10.8	9.4
3	2	18/7/04	100	0.7	15	385	413	10.8	9.4
3	3	18/7/04	75	0.7	15	445	413	8	9.4
3	4	18/7/04	50	0.7	15	rejected	413	10.8	9.4
3	5	18/7/04	25	0.7	15	405	413	11.5	9.4

The results for Runs 1 and 2 are shown in Figures 14 and 15. Note that the theoretical decomposition temperature is 413 degrees Centigrade for an infinitely long, perfectly insulated chamber. Both graphs show that this was nearly achieved at 10 mm downstream of the impingement point of the catalyst and HTP jets for both the unblended HSHP and RGHP runs. Indeed the temperature trends at the other stations are almost identical, which suggests that whatever is happening downstream of the 10 mm station it is definitely not due to the presence of stabilisers in the HSHP. A comparison of the indicated temperatures along the length of the chamber is presented in Figure 16 for all the Phase 3 runs. This will be commented on further in the next section.

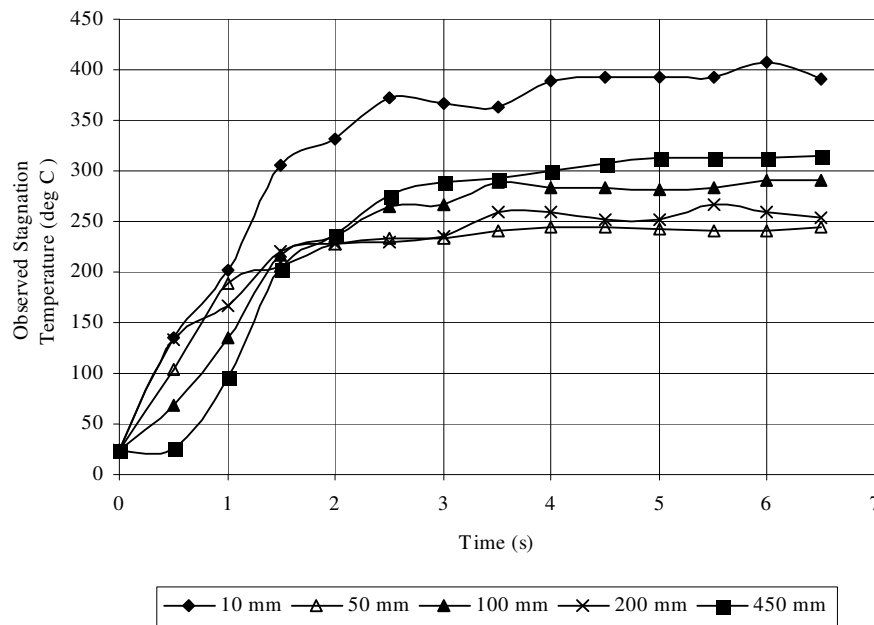


Figure 14. Phase 3/Run 1 Indicated Stagnation Temperatures for 0% RGHP (100% HSHP)

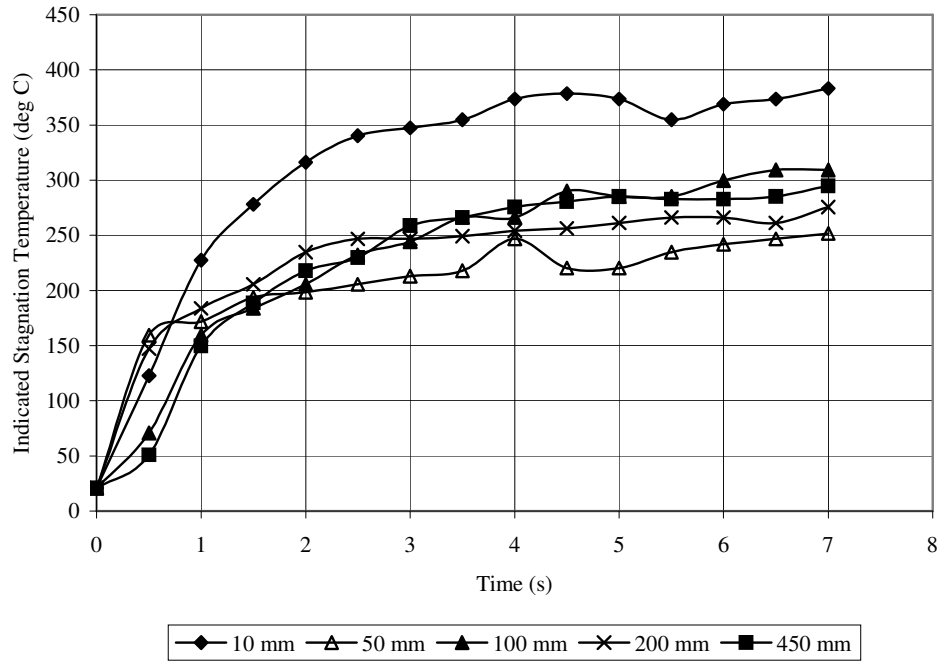


Figure 15 Phase 3/Run 2 Indicated Stagnation Temperatures for 100% RGHP (0% HSHP)

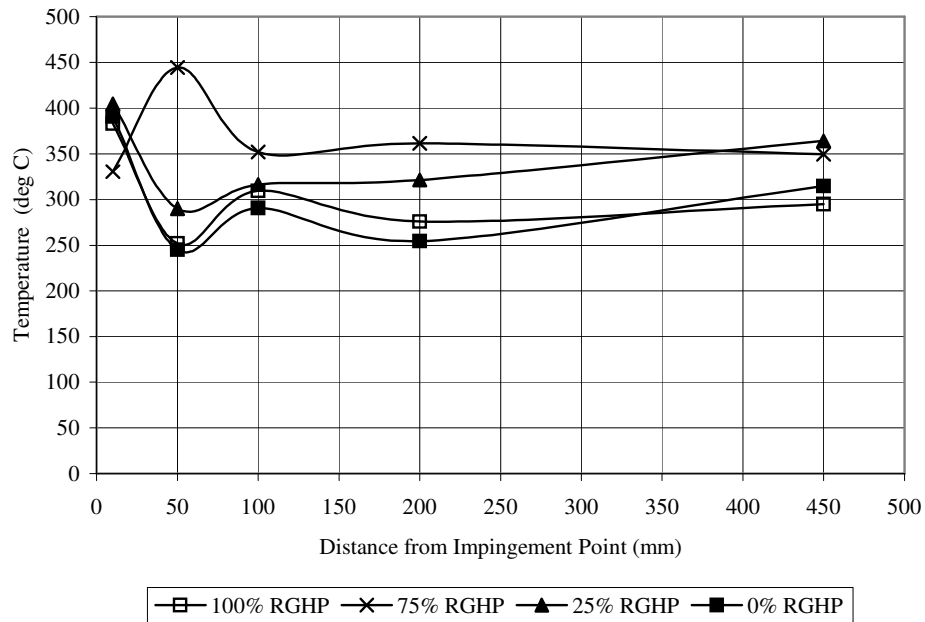


Figure 16. Phase 3 Indicated Temperatures along Reaction Chamber Axis

9. DISCUSSION

The earlier spray pattern tests suggested that although the bulk of the mixing appeared to take place at the point of impingement of the jets, the downstream shape of the flow retained a wake shadow of the cross associated with the four oxidant injectors being off the longitudinal axis of the chamber.

Moreover, the shape of the wake depended on the catalyst injector chosen. As a consequence it is conjectured that perfect mixing of the catalyst and HTP cannot be assumed to have taken place at the impingement point (datum), as is assumed in the thermo-chemical model.

In practice, a fair degree of mixing can be expected to take place in this region, but there is bound to be a fall-out of only very weakly catalysed fluid striking the internal wall of the reaction chamber some distance downstream of the impingement point (the spray pattern suggests about 25 mm). Given that the first thermocouple station is only 10 mm downstream of this datum, a certain degree of downstream cooling must be expected (analogous to film cooling in bi-propellant engines) since such partially catalysed fluid would still be reacting and would therefore continue to act as a coolant, with respect to the core catalysed material, until the reaction matured towards the nozzle. In such circumstances, because the shafts of the thermocouples were brazed directly into the wall reaction chamber, the indicated temperatures would be lowered downstream of the first station.

The above scenario is not applicable in the case of small catalyst injector diameters (low mixture ratios). Here, the flux of catalyst solution is too small to bring about a rapid reaction in the vicinity of the datum and instead brings about a gradual increase in stagnation temperature towards the nozzle. Figures 11-13 support this hypothesis. Neither is it likely to be applicable in the case of the largest catalyst injector. Here any fall-out might itself contain sufficient catalyst to bring about a very rapid reaction and hence its cooling effect would be minimal. Again, Figure 10 tends to support this account.

However, the fall-out effect is clearly seen for the 0.85 mm and 0.7 mm runs (corresponding to catalyst-to-HTP mixture ratios of 22% and 15% respectively) shown in Figures 11-13. Therefore, for the Phase 3 runs involving blends of HSHP and RGHP, all of which were conducted with the 0.7 mm catalyst injector, this cooling effect is to be expected beyond the first thermocouple station. The results shown in Figure 16 confirm this for the 100%, 25% and 0% RGHP cases. It is thought likely that for the case of 75% RGHP a partial blockage might have occurred in the catalyst injector leading to an effective orifice diameter that was somewhat smaller than 0.7 mm. This would have had the effect of causing the peak temperature to occur downstream of the first measuring station since the flow would still be very reactive.

Such blockage is thought to be due either to the nature of the material used to prepare the catalyst solution or to partial precipitation around the injector orifice caused by the heat of reaction. The sodium permanganate monohydrate was sourced from Sigma Aldrich (catalogue number 22,585-1) and is described as 97+% grade. It is quite possible therefore that foreign, insoluble matter might have caused the problem.

Although imperfect mixing was somewhat problematic for this study, nevertheless, runs 1 and 2 for Phase 3, which were undertaken with considerable attention to detail, demonstrate that the effect of very large amounts of stabilisers on the rate of homogeneous decomposition of hydrogen peroxide is insignificant. Both led to decomposition temperatures only slightly less than the figure of 413 degrees Centigrade, predicted for an infinitely long insulated chamber, within 10 mm of the point of impingement of the jets.

10. CONCLUSIONS

A homogeneous method of catalysis was adopted to investigate the decomposition of hydrogen peroxide operating in a mono-propellant mode at a nominal stagnation temperature of 400 degrees Centigrade. Guided by a simple thermo-chemical model of the process, a dedicated test rig was constructed with an emphasis on safe operation. The stagnation temperature along the axis of the reaction chamber was measured to compare, under controlled conditions, the decomposition characteristics of highly stabilised hydrogen peroxide (HSHP) with those of rocket grade hydrogen peroxide (RGHP).

The following conclusions were reached:

- The rig performed to specification and all tests were undertaken safely.

- Almost complete HSHP decomposition took place within 10 mm downstream of the jet impingement point for catalyst-to-HSHP mixture ratios greater than 15%.
- For the lowest catalyst-to-HSHP mixture ratio of 5%, complete decomposition took place somewhere between 100 and 450 mm downstream of the jet impingement point.
- For intermediate sizes of catalyst injector-hole diameter, some cooling was observed downstream of the first thermocouple station. This was attributed to the imperfect spray pattern observed.
- RGHP was prepared using semiconductor grade hydrogen peroxide to match the HSHP in peroxide concentration.
- Phosphorus levels in the HSHP were found to be one thousand times higher than those found in RGHP.
- Despite such high phosphorus content, there was a negligible difference in the indicated stagnation temperatures between the HSHP and RGHP propellants.
- For the chosen catalyst injector, almost complete decomposition was achieved 10 mm downstream of the jet impingement point for both the HSHP and RGHP .
- More perfect mixing in the injector region would probably reduce the catalyst loading factor.
- More perfect mixing in the injector region would also reduce the wall cooling and so increase the decomposition temperature further downstream.

11. RECOMMENDATIONS FOR FURTHER WORK

Future research into the feasibility of using HSHP for future applications can be divided into two categories. The first category adopts the cautionary stance that more work is needed to confirm the tentative conclusion drawn in this report that HSHP can be made to decompose as quickly as RGHP, when used with an effective liquid catalyst. The second category assumes that this conclusion is sound and does not warrant further confirmation.

The following recommendations fall into the first category:

- Investigate the transient build-up of pressure within a bespoke bomb calorimeter associated with the impulsive combination of a catalyst with either RGHP or HSHP. A delayed pressure rise in the case of HSHP would indicate a slower rate of decomposition.
- Repeat the investigation described herein but with a splash plate fitted to the injector face to improve mixing.
- Perform runs with a lower catalyst loading factor to exaggerate the differences in decomposition rates between the two types of HTP. To achieve this, foreign matter in the catalyst solution would need to be removed using a very fine stainless steel filter.

The following recommendations fall into the second category:

- Experiments should be performed with higher concentrations of HSHP in order to raise the decomposition temperature to that needed for auto-ignition of a fuel.
- Explore the use of alternative liquid catalysts or reactants for decomposing highly stabilised hydrogen peroxide.
- Explore the feasibility of using rapidly decomposed HSHP in conjunction with a range of fuels. The fuels should be chosen such as to maximise the likelihood of auto-ignition, thus obviating the need for an ignition system.
- Investigate the storage properties of HSHP under controlled conditions.

12. ACKNOWLEDGEMENTS

This work was part-sponsored by DELTACAT Limited, UK, and part-sponsored by the Air Force Office of Scientific Research, Air Force Material Command, USAF, under grant number FA8655-04-1-

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The author is grateful for the able assistance of Dr Graham Roberts, Paul Chandler, John Grayson and Joseph Holdsworth (all of the University of Southampton, UK). The trace elements in the samples of HTP were analysed by John Wilson at the Environmental Analysis Laboratories in Hastings, UK.

Disclaimer: The views and conclusions contained herein are those of the author and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Air Force Office of Scientific Research or the US Government.

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Appendix - Procedures adopted for live tests

The procedures used during the live tests were the result of a careful assessment of risk to personnel at each stage of the preparations. Correct interpretation of the procedures was facilitated by reference to Figure A1, which provides details of the rig, including the reference numbers used for the various valves and end-stops. Laminated copies of Figure A1 were available at both the test stand and the control centre.

The following convention was used to describe the valves, depending on their function: MV denotes a manually operated valve, SV denotes a direct acting solenoid-actuated valve and PL refers to plugs that must be removed for filling and replaced for firing.

The main nitrogen regulator (not pictured) provides the required system pressure from the nitrogen gas bottle and during actual runs was set to a gauge pressure of 15 bar gauge. The regulator can provide pressures up to 28 bar and contains Stainless Steel diaphragms for high pressure service.

The first major component at the top of the schematic is MV1. This valve is a crucial safety valve that manually shuts off the nitrogen supply in the event of SV2 failing. The valve itself is a standard ½ inch ball valve and identical valves are used for MV2 and MV3, which permit the flow of nitrogen into the catalyst and oxidant tanks respectively. MV2 and MV3 were installed to prevent the possibility of the hydrogen peroxide and NaMnO₄ coming into contact. Detailed operating procedures determining the status of all the valves ensure that the engine is completely shutdown following use.

Connected to MV1 is SV2, which remotely allows the flow of nitrogen into the system. The valve allows rig personnel to retreat to a safe distance, having opened MV1. The solenoid valves are all direct acting and have Viton seals installed. The solenoid valves are either normally open or normally closed depending on the purpose. For example, SV1 is a vent valve that is normally open to allow any gases to escape, whereas the natural state of SV2 is closed.

The pipes and individual compression fittings are all manufactured from 316 grade stainless steel and most are single ferrule fittings to reduce the cost of the engine. The system was pressure-tested and checked for leaks prior to commissioning (the hydrogen peroxide tank was tested to 30 bar). The threads of the joints were coated with blue Hylomar, which is a polyester polyol-based compound that has a non-hardening formula to maintain seal integrity and easy assembly or disassembly even when subjected to thermal distortion and vibration.

The gauges used to measure the chamber and tank pressures are glycerine free to prevent any hazardous reaction with the HSHP and display pressure readings up to 30 bar. It was also decided at the design stage to incorporate a safety relief valve as an additional safety feature. The relief valve prevents potentially hazardous increases in gas pressures. The relief valve is opened by the pressure overcoming the spring stiffness, which was preset to a pressure of 23 bar.

LED indicators were connected to the electrical system, which allowed remote monitoring of the status of each solenoid valve using a video camera linked to the control centre. This was located approximately 60m away from the test stand. The camera also captured the tank pressure gauge reading and the status of the low-pressure nitrogen regulator as illustrated in figure 8-3. The low-pressure regulator provides a step down in nitrogen pressure from 15 to 8 bar to open the actuators of the pneumatically operated ball valves discussed below. Solenoid valves SV3 and SV4 control the flow of nitrogen to these valves, whilst SV5 and SV6 vent each line independently to allow the pneumatically actuated valves to close. The electrical connection of these valves was such that when SV3 and SV4 were open, SV5 and SV6 were closed.

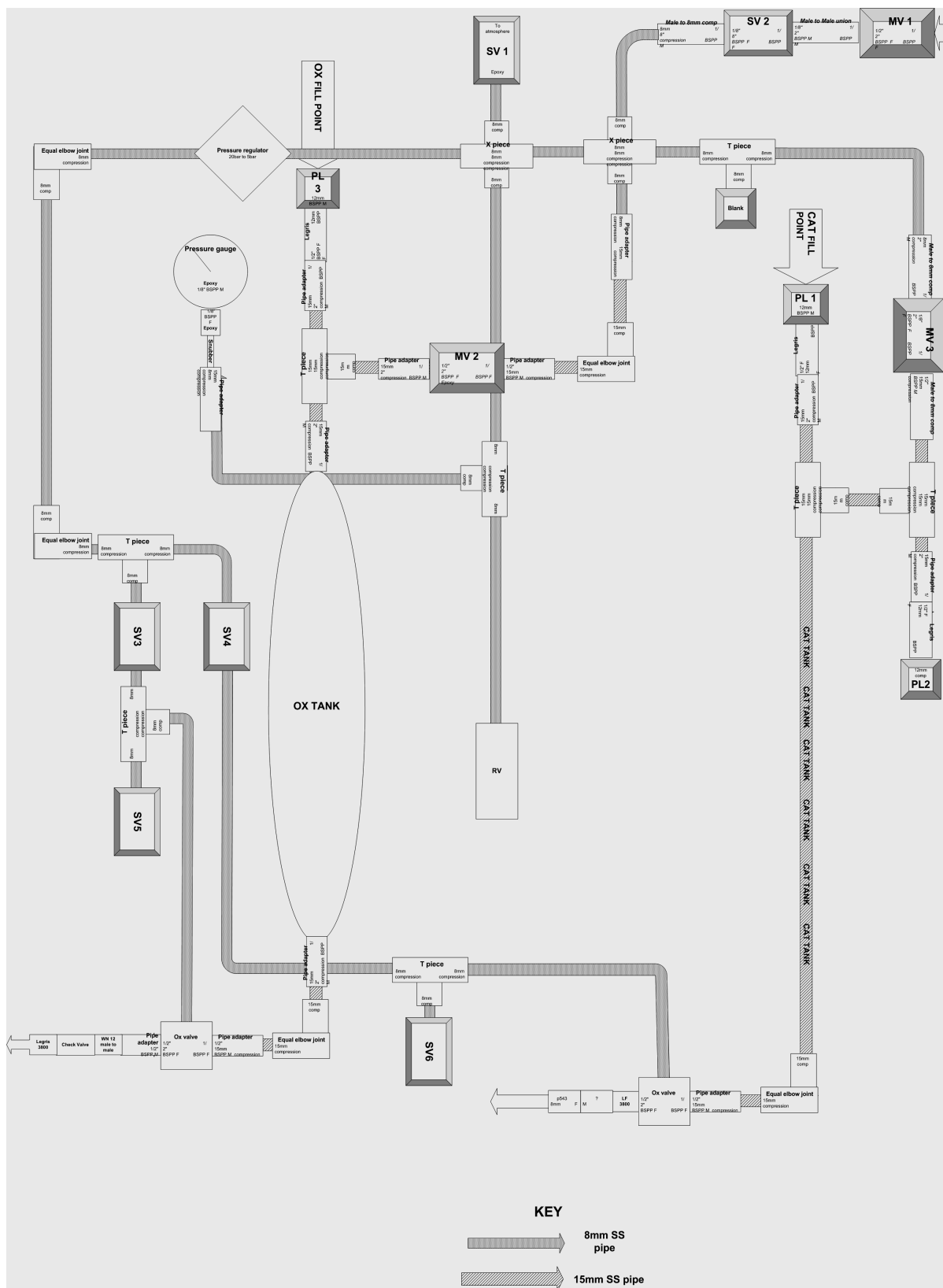


Figure A1. Schematic of Test-Rig

The hydrogen peroxide valve is remotely operated through various solenoid valves and presented certain challenges. The valve had to be able to allow any decomposition gases within the valve itself to escape. This situation could occur if any residual peroxide remained trapped in the valve over long periods of time following the flushing operation. Solenoid valves and other candidate valves were discounted due to the relation between the pressure ratings decreasing with increasing valve orifice size. The selection of a pneumatically actuated three piece full-bore valve provided the high pressure rating and increased orifice size that was required to achieve the desired mass flow rate. The valve is manufactured from 316 stainless steel and has PTFE seals and seats to prevent degradation of the seals by the aggressive action of high concentration HSHP during operation. The ball was custom-drilled to allow the ball to vent once the valve was closed after a run. The valve can also be easily stripped to allow seal replacement if necessary and if required the actuators can be manually turned to open the valve in the absence of the nitrogen supply.

A similar arrangement was used for the catalyst line. The main differences were a reduced orifice size, due to the reduction in mass flow rate relative to the HSHP valve, and the absence of the vent hole.

Downstream of the HSHP pneumatic valve is a non-return check valve that prevents any surge in pressure forcing HSHP in the wrong direction. The check valve prevents a possible catastrophic failure in the event of depressurisation of the rig (for any reason) whilst the reaction is occurring in the decomposition chamber. The reaction could potentially force HSHP and catalyst up the feed line into the HSHP tank, which would cause the decomposition reaction to occur in the HSHP tank and would consequently lead to a large rapid pressure increase and failure. The non-return valve has a cracking pressure of 0.2 bar and is manufactured from 316 Stainless Steel with PTFE/viton body seals and viton poppet seals.

Following the non-return valve are push-fit attachments that allow the easy removal of the flexible feed lines that connect the plumbing board and the injector manifold, which is connected to the decomposition chamber.

Note that there is also provision on the plumbing board for a fuel tank to make the rocket a bi-propellant engine for future work.

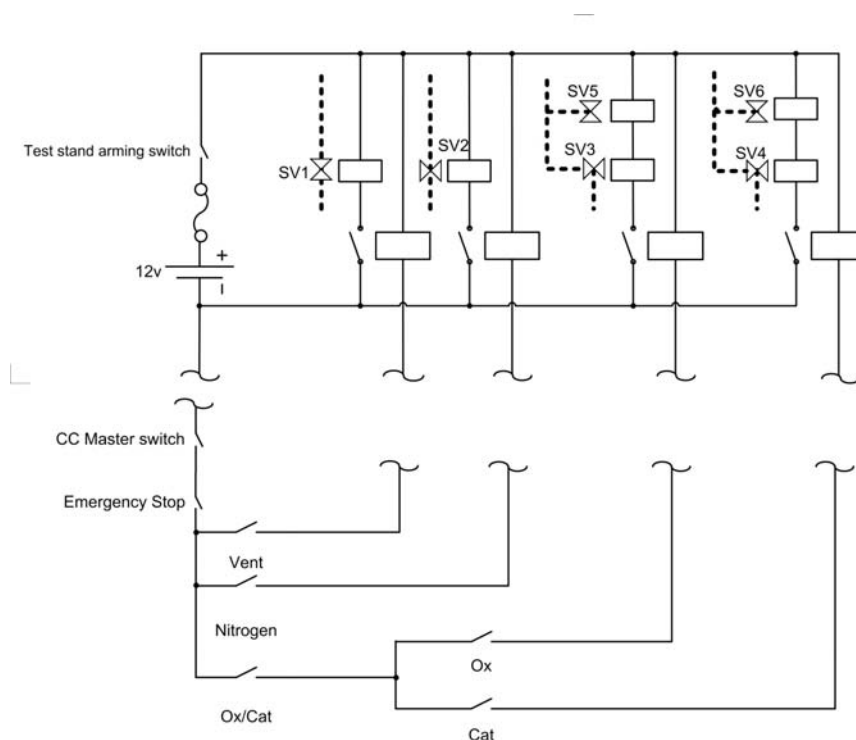


Figure A2. Wiring diagram

Figure A2 shows the electrical wiring associated with operation of the solenoid valves. The design called for a remote control centre with relevant switches to activate the required solenoid valves. This remote control centre was connected to the rig via a 100m multi-core cable.

Due to the resistance in the 100m connection-cable, the voltage drop across it was too high and this inhibited direct activation of the solenoid valves. This led to the installation of relays into the circuit. The coil in the relay was activated by the Master Control Panel (MCP). This in turn closed the switch in the relay and activated the solenoid valve. The power to activate the solenoid valves came from two 12v lead-acid batteries wired in parallel. These are located next to the rig in the test stand. The catalyst and peroxide valves are activated by the ox/cat combination switch. This was to ensure both the catalyst and H_2O_2 were released into the chamber at the same time. Prior to activating this dual firing switch, the catalyst and peroxide lines were 'armed' in a pre-determined sequence.

The safety features of the final rig operation are as follows:

1. A master key to power on and off the master control panel
2. A second master key to power on and off the rig. Both keys had to be in and turned to the 'on' position for any of the valves to work.
3. A detailed countdown system, including individual arming of the hydraulic lines.
4. An emergency stop button located on the Master Control Panel which, when pressed, returns all solenoid valves to their original state and vents the rig of pressure.

The procedures for each phase of testing and firing were derived after a careful study of the system. Dry runs were performed to test the procedures, which were then refined in the light of experience. The procedures were based on a number of spreadsheets, which represent the state of all plumbing controls. An example is given in Table A1 for the case of the actual firing of the rig. The columns represent the sequence in time and the rows represent the controls (O stands for OPEN and C stands for CLOSED).

From the spreadsheet, each valve sequence was converted into a text command. Over time, a complete text sequence for the valve commands was developed to cover all phases of testing and firing.

Once the rig was constructed, each stage of the procedures was analysed in detail to make any refinements or additions by performing a walk-through at the rig. At this stage all the non-valve related commands were added (such as observing LED status and gauge readings).

As testing approached further additions were made. One of these additions was a valve status table at the start of each sequence. This meant the text procedures would stand-alone from the spreadsheet. Complete shutdown procedures were added to the relevant sections to ensure the rig returned to a safe state at the end of testing. These ensured that no residual chemicals or nitrogen gas remained anywhere in the system but also took precautions just in case to prevent any slow build up of pressure from naturally decomposing HTP.

		Running							1	2	3	4	5	6	7
MV1	O	O	O	O	O	O	C,1	1 Original State							
MV2	O	O	O	O	O	O	C,3	2 Check Vent LED							
MV3	O	O	O	O	O	O	C,6	3 Check N2 LED							
SV1	O	C,1	C	C	C	O,2	O	4 Check Ox & Cat LED							
SV2	C	C	O,1	O	O	C,1	C	5 N2 Purge for 5s							
SV3	C	C	C	O,1	O	O	C,7	6 Vent, Check Ox & Cat valves are open then flush immediately after rig declared safe							
		SV4	C	C	C	O,1	O	O	C,8						
		SV5	O	O	O	C,1	C	C	O						
		SV6	O	O	O	C,1	C	C	O						
		PL1	C	C	C	C	C	C	O,5						
		PL2	C	C	C	C	C	C	O,4						
		PL3	C	C	C	C	C	C	O,2						
		PL4	O	O	O	O	O	O	O						

Table A1 Procedures Spreadsheet

The following sections provide details of the various phases during a test period.

1 Pressure Testing

1. Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	C	C	C	O

- ☐ Check main nitrogen bottle valve is closed
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Set low pressure nitrogen regulator to zero pressure (Turn fully anti-clockwise)
- ☐ Check all switches are off, valve closed (electrically inactive)
- ☐ Connect battery to power rig
- ☐ Turn rig master key switch on
- ☐ On radio command, insert control panel key and turn on
- ☐ Operate each switch independently, checking functionality and LED's
- ☐ Turn off both master key switches
 - ☐ Control Panel
 - ☐ Rig
- ☐ Check all control panel switches are up (electrically inactive)
- 2. Testing the Solenoid side
 - ☐ Close main nitrogen Regulator (Turn fully Anti-clockwise)
 - ☐ Rig personnel to put on PPE (Goggles)
 - ☐ Open main nitrogen bottle valve
 - ☐ Set main nitrogen regulator to required pressure (Turning clockwise)
 - ☐ Activate rig key switch
 - ☐ Open Manual Valve 1 (MV1 - Nitrogen)
 - ☐ Short retreat
 - ☐ On radio command, insert control panel key and turn on
 - ☐ Close Solenoid Valve 1 (SV1 - Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Check for leaks
 - ☐ Set low pressure nitrogen regulator to 6 bar (Turning clockwise)
 - ☐ Mark the position of the low pressure regulator
 - ☐ Await instructions for checking actuator valves
- 3. Venting
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Check LED's
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ For complete vent, move to sequence 10
- 4. Testing Catalyst Side, Top End
 - ☐ Open Manual Valve 3 (MV3 – Catalyst)
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Check for leaks
- 5. Venting
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
 - ☐ For complete vent, move to sequence 10
- 6. Testing Ox Side, Top End
 - ☐ Open Manual Valve 2 (MV2 – Ox)

- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat
- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Check pressure gauge on monitor
- ☐ Check for leaks

7. Venting

- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Open Solenoid Valve 1 (SV1 – Vent)
- ☐ Check pressure gauge on monitor (drops to zero)
- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Open Manual Valve 3 (MV3 – Catalyst)
- ☐ For complete vent, move to sequence 10

8. Full System Test

- ☐ Insert Chamber Plug (PL4 – Chamber)
- ☐ Check position of low pressure regulator
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat
- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Set Ox/Cat combination switch to valve open (Armed)
- ☐ Open Solenoid Valve 3 (SV3 – Ox)
- ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Check pressure gauge on monitor
- ☐ Check for leaks

9. Venting

- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Open Solenoid Valve 1 (SV1 – Vent)
- ☐ Check pressure gauge on monitor (drops to zero)
- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Set Ox/Cat combination switch to valve closed (Disarmed)
- ☐ Close Solenoid Valve 3 (SV3 – Ox)
- ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Close Manual Valve 3 (MV3 – Catalyst)
- ☐ Close Manual Valve 2 (MV2 – Ox)

10. Complete Venting including nitrogen hose

- ☐ Close main nitrogen bottle valve
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Monitor pressure gauge (drops to zero)

11. Shutdown and Preparation for Filling

- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Remove Chamber Plug (PL4 – Chamber)
- ☐ Remove Ox Plug (PL3)
- ☐ Check bottle pressure gauges are zero
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Turn off control panel key switch
- ☐ Turn off rig key switch
- ☐ Disconnect battery
- ☐ Check control panel switches are all up (Electrically inactive)

2 Filling

1. Valve and Switch functionality check

- ☐ Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	C	C	C	O

- ☐ Check main nitrogen bottle valve is closed
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Check all switches are off, valve closed (electrically inactive)
- ☐ Connect battery to power rig
- ☐ Turn rig master key switch on
- ☐ On radio command, insert control panel key and turn on
- ☐ Operate each switch independently, checking functionality and LED's
- ☐ Turn off both master key switches
 - ☐ Control Panel
 - ☐ Rig
- ☐ Check all control panel switches are up (electrically inactive)
- ☐ Check main nitrogen bottle valve is closed
- ☐ Check main nitrogen regulator is closed (Turn fully Anti-clockwise)
- ☐ Check all switches are off, valve closed (electrically inactive)
- ☐ Rig personnel to put on PPE (Goggles)

2. Filling the Catalyst Tank

- ☐ Remove Upper Catalyst Plug (PL2)
- ☐ Remove Lower Catalyst Plug (PL1 – Overflow)
- ☐ Position overflow receptacle for catalyst solution
- ☐ Fill Catalyst Tank

3. Close Up of Catalyst

- ☐ Insert Upper Catalyst Plug (PL2)
- ☐ Insert Lower Catalyst Plug (PL1 – Overflow)
- ☐ Wash hands, Change gloves

4. Filling the Ox Tank

- ☐ Remove Ox Plug (PL3)
- ☐ Check PPE is on
- ☐ Fill Ox Tank

5. Preparation for Firing

- ☐ Insert Ox Plug (PL3)
- ☐ Open Manual Valve 2 (MV2 – Ox)
- ☐ Check Video Cameras – Gauges and plume
- ☐ Place radio next to Camera microphones
- ☐ Check cold junction temperature
- ☐ Check Manual Valve 1 is closed (MV1 – Nitrogen)
- ☐ Open main nitrogen bottle valve
- ☐ Set main nitrogen regulator to required pressure (Turning clockwise)
- ☐ Check position of low pressure regulator
- ☐ Activate rig key switch
- ☐ Start Data Acquisition system – see data acquisition.doc

6. Final Priming Sequence

- ☐ Set Digital Voltmeter's to 200mV
- ☐ Turn on all cameras to record
- ☐ Close Manual Valve 2 (MV2 – Ox)
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Short Retreat

3 Running

1. Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
O	O	C	O	C	C	C	O	O	C	C	C	O

- ☐ All mobile phones to be switched off
- ☐ On command insert control panel key switch and turn on
- ☐ Await clearance from field
- 2. Sealing the system for pressure adjustment
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check Vent LED on monitor
- 3. Releasing Nitrogen
 - ☐ Check Ox/Cat combination switch is disarmed (Valve closed)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Check Nitrogen LED on monitor
 - ☐ Team leader approaches
- 4. Fine adjustment of supplied pressure
 - ☐ Adjust main nitrogen regulator until main gauge reads 15 bar
 - ☐ Short retreat
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (Drops to zero)
 - ☐ Approach rig
 - ☐ Open Manual Valve 2 (MV2 – Ox)
 - ☐ Open Manual Valve 3 (MV3 – Cat)
 - ☐ Full retreat
- 5. Sealing the system for firing
 - ☐ Await clearance from field
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check Vent LED on monitor
- 6. Releasing Nitrogen
 - ☐ Check Ox/Cat combination switch is disarmed (Valve closed)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Check Nitrogen LED on monitor
- 7. Firing
 - ☐ Open Solenoid Valve 3 (SV3 – Ox)
 - ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Check Ox and Cat LED on monitor (Remain Unlit)
 - ☐ Press radio transmit button to record countdown
 - ☐ Set Ox/Cat combination switch to valve open (Armed – Ox/Cat LED's Lit)
- 8. Purging
 - ☐ Maintain Nitrogen for 5 seconds after complete exhaust of ox and catalyst
- 9. Venting
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Train binoculars on rig to check for fire
 - ☐ Approach rig
- 10. Shutdown and preparation for flushing
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close main nitrogen bottle valve
 - ☐ Rig personnel to wear PPE
 - ☐ Remove Ox Plug (PL3)
 - ☐ Close Manual Valve 2 (MV2 – Ox)
 - ☐ Remove Upper Catalyst Plug (PL2)
 - ☐ Remove Lower Catalyst Plug (PL1)
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
 - ☐ Close Solenoid Valve 3 (SV3 – Ox)

- ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Set Ox/Cat combination switch to valve closed (Disarmed)

4 Flushing (Both Lines Simultaneously)

1. Check original state of all valves table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	O	O	O	O

2. Filling, Manual release and retreat

- ☐ Position Catalyst overflow receptacle
- ☐ Fill both tanks with deionised water
- ☐ Insert Lower Catalyst Plug (PL1)
- ☐ Insert Upper Catalyst Plug (PL2)
- ☐ Insert Ox Plug (PL3)
- ☐ Open main nitrogen bottle valve
- ☐ Check main nitrogen regulator setting
- ☐ Check low pressure regulator position
- ☐ Open Manual Valve 2 (MV2 – Ox)
- ☐ Open Manual Valve 3 (MV3 – Catalyst)
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat

3. Flushing

- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Check pressure gauge on monitor
- ☐ Open Solenoid Valve 3 (SV3 – Ox)
- ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Set Ox/Cat combination switch to valve open (Armed)
- ☐ Pulse Ox/Cat combination switch in 1 second intervals

4. Venting and Electrical Shutdown

- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Open Solenoid Valve 1 (SV1 – Vent)
- ☐ Check pressure gauge on monitor (drops to zero)
- ☐ Close Solenoid Valve 3 (SV3 – Ox)
- ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Set Ox/Cat combination switch to valve closed (Disarmed)

5. Approach and Make safe

- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Close main nitrogen bottle valve
- ☐ Close Manual Valve 2 (MV2 – Ox)
- ☐ Close Manual Valve 3 (MV3 – Catalyst)
- ☐ Remove Ox Plug (PL3)
- ☐ After sufficient repetitions move to Stage 7
- ☐ If more runs are needed continue to Stage 6

6. Repeat flushing

- ☐ Remove Lower Catalyst Plug (PL1)
- ☐ Remove Upper Catalyst Plug (PL2)
- ☐ From here, return to Stage 2

7. Complete Venting and Shutdown

- ☐ Loosen Upper Catalyst Plug (PL2)
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Monitor pressure gauge (drops to zero)
- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Check bottle pressure gauges are zero
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)

- ☐ Turn off control panel key switch
- ☐ Turn off rig key switch
- ☐ Check data acquisition system procedure for data collection
- ☐ Disconnect battery
- ☐ Check control panel switches are all up (Electrically inactive)

5 Repeated Flushing of Catalyst Tank (follows from Running)

1. Check original state of all valves against check sheet

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	O	O	O	O

2. Filling, Manual Release and Retreat

- ☐ Fill Catalyst Tank with deionised water
- ☐ Insert Lower Catalyst Plug (PL1)
- ☐ Insert Upper Catalyst Plug (PL2)
- ☐ Check low pressure regulator position
- ☐ Open Manual Valve 3 (MV3 – Catalyst)
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat

3. Flushing

- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Check pressure gauge on monitor
- ☐ Set Ox/Cat combination switch to valve open (Armed)
- ☐ Open Solenoid Valve 4 (SV4 – Catalyst)

4. Venting and Electrical Shutdown

- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Open Solenoid Valve 1 (SV1 – Vent)
- ☐ Check pressure gauge on monitor (drops to zero)
- ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Set Ox/Cat combination switch to valve closed (Disarmed)

5. Approach and make safe

- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ After sufficient flushing move to Stage 8 for shutdown
- ☐ Remove Lower Catalyst Plug (PL1)
- ☐ Remove Upper Catalyst Plug (PL2)

6. Filling, Manual Release and Retreat

- ☐ Fill Catalyst Tank with deionised water
- ☐ Insert Lower Catalyst Plug (PL1)
- ☐ Insert Upper Catalyst Plug (PL2)
- ☐ Check low pressure regulator position
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat

7. Repeated Flushing (Return to Stage 3)

8. Complete Venting and Shutdown

- ☐ Close main nitrogen bottle valve
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Monitor pressure gauge (drops to zero)
- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
- ☐ Approach rig
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Check bottle pressure gauges are zero
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Turn off control panel key switch
- ☐ Turn off rig key switch
- ☐ Disconnect battery
- ☐ Check control panel switches are all up (Electrically inactive)

6 Repeated Flushing of Ox Tank (follows from Stage 5 of Repeated Catalyst Tank flushing)

1. Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	O	O	C	C	C	O	O	C	C	O	O

2. Filling
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
 - ☐ Open Manual Valve 2 (MV2 – Ox)
 - ☐ Fill Ox Tank with deionised water
3. Manual Release and Retreat
 - ☐ Insert Ox Plug (PL3)
 - ☐ Check low pressure regulator position
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
4. Flushing
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Set Ox/Cat combination switch to valve open (Armed)
 - ☐ Open Solenoid Valve 3 (SV3 – Ox)
5. Venting and Electrical Shutdown
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Close Solenoid Valve 3 (SV3 – Ox)
 - ☐ Set Ox/Cat combination switch to valve closed (Disarmed)
6. Approach and Make safe
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ After sufficient flushing move to Stage 8 for shutdown
 - ☐ Remove Ox Plug (PL3)
 - ☐ Fill Ox Tank with deionised water
7. Repeated Flushing (Return to stage 3)
8. Complete Venting and Shutdown
 - ☐ Close main nitrogen bottle valve
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Monitor pressure gauge (drops to zero)
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Check bottle pressure gauges are zero
 - ☐ Close main nitrogen regulator (Turn fully anti-clockwise)
 - ☐ Turn off control panel key switch
 - ☐ Turn off rig key switch
 - ☐ Disconnect battery
 - ☐ Check control panel switches are all up (Electrically inactive)

7 Discharge Coefficient Determination (Individual lines - No venting between)

1. Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	C	C	C	O

- ☐ Check main nitrogen bottle valve is closed
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Check all switches are off, valve closed (electrically inactive)
- ☐ Connect battery to power rig
- ☐ Turn rig master key switch on
- ☐ On radio command, insert control panel key and turn on
- ☐ Operate each switch independently, checking functionality and LED's
- ☐ Turn off both master key switches
- ☐ Check all control panel switches are up (electrically inactive)
- ☐ Check main nitrogen bottle valve is closed
- ☐ Check main nitrogen regulator is closed (Turn fully Anti-clockwise)
- ☐ Check all switches are off, valve closed (electrically inactive)

2. Filling both tanks

- ☐ Position Catalyst overflow receptacle
- ☐ Remove Lower Catalyst Plug (PL1)
- ☐ Remove Upper Catalyst Plug (PL2)
- ☐ Fill Catalyst Tank with measured amount of deionised water
- ☐ Insert Lower Catalyst Plug (PL1)
- ☐ Insert Upper Catalyst Plug (PL2)
- ☐ Remove Ox Plug (PL3)
- ☐ Fill Ox Tank with measured amount of deionised water
- ☐ Insert Ox Plug (PL3)
- ☐ Close main nitrogen regulator (Turn fully anti-clockwise)
- ☐ Open main nitrogen bottle valve
- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Open Manual Valve 1 slowly (MV1 – Nitrogen)
- ☐ Adjust main nitrogen regulator to required pressure (Monitor main gauge)
- ☐ Check low pressure regulator position
- ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Close Manual Valve 1 (MV1 – Nitrogen)
- ☐ Stand back from rig
- ☐ Open Solenoid Valve 1 (SV1 – Vent)
- ☐ Check pressure gauge on monitor (drops to zero)
- ☐ Approach rig
- ☐ Open Manual Valve 3 (MV3 – Catalyst)
- ☐ Open Manual Valve 2 (MV2 – Ox)
- ☐ Check video cameras – Gauges and spray plume
- ☐ Open Manual Valve 1 (MV1 – Nitrogen)
- ☐ Retreat

3. Timed Firings

- ☐ Close Solenoid Valve 1 (SV1 – Vent)
- ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
- ☐ Check pressure gauge on monitor
- ☐ Set Ox/Cat combination switch to valve open (Armed)
- ☐ Use TV monitor/Stopwatch to record flow rate
- ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Measure test duration until all water is released
- ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
- ☐ Check Ox/Cat combination switch is valve open (Armed)
- ☐ Use TV monitor/Stopwatch to record flow rate
- ☐ Open Solenoid Valve 3 (SV3 – Ox)
- ☐ Measure Test duration until all water is released
- ☐ Open Solenoid Valve 4 (SV4 – Catalyst)

- ☐ Maintain nitrogen for 5s to purge the system of water
- 4. Venting and Electrical Shutdown
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Close Solenoid Valve 3 (SV3 – Ox)
 - ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Set Ox/Cat combination switch to valve open (Disarmed)
- 5. Approach and Make safe
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close Manual Valve 2 (MV2 – Ox)
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
- 6. Testing both lines together
 - ☐ Position Catalyst overflow receptacle
 - ☐ Remove Lower Catalyst Plug (PL1)
 - ☐ Remove Upper Catalyst Plug (PL2)
 - ☐ Fill Catalyst Tank with measured amount of deionised water
 - ☐ Insert Lower Catalyst Plug (PL1)
 - ☐ Insert Upper Catalyst Plug (PL2)
 - ☐ Remove Ox Plug (PL3)
 - ☐ Fill Ox Tank with measured amount of deionised water
 - ☐ Insert Ox Plug (PL3)
 - ☐ Check low pressure regulator position
 - ☐ Open Manual Valve 3 (MV3 – Catalyst)
 - ☐ Open Manual Valve 2 (MV2 – Ox)
 - ☐ Check video cameras – Gauges and spray plume
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
- 7. Timed Firings
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Open Solenoid Valve 3 (SV3 – Ox)
 - ☐ Set Ox/Cat combination switch to valve open (Armed)
 - ☐ Use TV monitor/Stopwatch to record flow rate
 - ☐ Measure test duration until all water is released
 - ☐ Maintain nitrogen for 5s to purge the system of water
- 8. Venting and Electrical Shutdown
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Close Solenoid Valve 3 (SV3 – Ox)
 - ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Set Ox/Cat combination switch to valve open (Disarmed)
- 9. Approach and Make safe
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close Manual Valve 2 (MV2 – Ox)
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
- 10. Complete Venting and Shutdown
 - ☐ Close main nitrogen bottle valve
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Monitor pressure gauge (drops to zero)
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)

- ☐ Check bottle pressure gauges are zero
- ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
- ☐ Turn off control panel key switch
- ☐ Turn off rig key switch
- ☐ Disconnect battery
- ☐ Check control panel switches are all up (Electrically inactive)

8 Discharge Coefficient Determination (Individual lines with venting between)

1. Check original state of all valves against table below

MV1	MV2	MV3	SV1	SV2	SV3	SV4	SV5	SV6	PL1	PL2	PL3	PL4
C	C	C	O	C	C	C	O	O	C	C	C	O

2. Testing Catalyst Line
 - ☐ Remove Lower Catalyst Plug (PL1)
 - ☐ Remove Upper Catalyst Plug (PL2)
 - ☐ Fill Catalyst Tank with measured amount of deionised water
3. Manual Release and Retreat
 - ☐ Insert Lower Catalyst Plug (PL1)
 - ☐ Insert Upper Catalyst Plug (PL2)
 - ☐ Set main nitrogen regulator fully anti-clockwise
 - ☐ Open main nitrogen bottle valve
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Manual Valve 1 slowly (MV1 – Nitrogen)
 - ☐ Adjust main nitrogen regulator to required pressure (Monitor main gauge)
 - ☐ Check low pressure regulator position
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Stand back from rig
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Manual Valve 3 (MV3 – Catalyst)
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
4. Timed Firing of Catalyst Line
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Set Ox/Cat combination switch to valve open (Armed)
 - ☐ Use TV monitor/Stopwatch to record flow rate
 - ☐ Open Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Measure test duration until all water is released
 - ☐ Maintain nitrogen for 5s to purge the system of water
5. Venting and Electrical Shutdown
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Close Solenoid Valve 4 (SV4 – Catalyst)
 - ☐ Set Ox/Cat combination switch to valve closed (Disarmed)
6. Approach and Make safe
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close Manual Valve 3 (MV3 – Catalyst)
7. Testing Ox Line
 - ☐ Remove Ox Plug (PL3)
 - ☐ Fill Ox Tank with measured amount of deionised water
8. Manual Release and Retreat
 - ☐ Insert Ox Plug (PL3)
 - ☐ Open Manual Valve 2 (MV2 – Ox)
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
9. Timed Firing of Ox Line
 - ☐ Close Solenoid Valve 1 (SV1 – Vent)
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Check pressure gauge on monitor
 - ☐ Set Ox/Cat combination switch to valve open (Armed)

- ☐ Use TV monitor/Stopwatch to record flow rate
- ☐ Open Solenoid Valve 3 (SV3 – Ox)
- ☐ Measure Test duration until all water is released
- 10. Venting and Electrical Shutdown
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Open Solenoid Valve 1 (SV1 – Vent)
 - ☐ Check pressure gauge on monitor (drops to zero)
 - ☐ Close Solenoid Valve 3 (SV3 – Ox)
 - ☐ Set Ox/Cat combination switch to valve open (Disarmed)
- 11. Approach and Make safe
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Close Manual Valve 2 (MV2 – Ox)
- 12. Complete Venting and Shutdown
 - ☐ Close main nitrogen bottle valve
 - ☐ Open Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Retreat
 - ☐ Open Solenoid Valve 2 (SV2 – Nitrogen)
 - ☐ Monitor pressure gauge (drops to zero)
 - ☐ Close Solenoid Valve 2 (SV2 – Nitrogen) at pressure zero
 - ☐ Approach rig
 - ☐ Close Manual Valve 1 (MV1 – Nitrogen)
 - ☐ Check bottle pressure gauges are zero
 - ☐ Close main nitrogen regulator (Turn fully Anti-clockwise)
 - ☐ Turn off control panel key switch
 - ☐ Turn off rig key switch
 - ☐ Disconnect battery
 - ☐ Check control panel switches are all up (Electrically inactive)